Workshop on Purity and Dispersion Measurement Issues of Single Wall Carbon Nanotubes (SWCNTs)

NIST, Gaithersburg, Maryland
May 27-29, 2003

Executive Summary

A workshop organized jointly by the National Aeronautics and Space Administration, Lyndon B. Johnson Space Center (NASA/JSC) and the National Institute of Standards and Technology (NIST) was held on May 27-29, 2003 at NIST in Gaithersburg, MD. In attendance were 68 participants, representing 11 private corporations, 19 universities, and 9 government agencies. The primary purpose of the workshop was to bring together leading researchers in the field of single wall carbon nanotubes (SWCNTs) to discuss and prioritize measurement needs relative to nanotube purity and dispersion.

The topics of purity and dispersion were chosen as focus areas because of their critical importance for future development of SWCNT applications. Currently, a variety of measurement techniques employing significant differences in test methodology are utilized for purity and dispersion assessment resulting in unacceptable uncertainties in data interpretation from one laboratory to another. For this reason, comparison of different SWCNT materials is extremely difficult.

To address the above measurement challenges, the organizing committee invited 23 speakers, and developed an agenda that encouraged active participation from attendees. Breakout sessions addressing both workshop topics were held to foster open discussion and to invite consensus regarding best techniques and measurement methods. A final panel discussion led to recommendations for future work and to plans for developing documentation of existing techniques. The agenda, as well as speaker and poster session abstracts, is included in the appendices.

The purity of single walled carbon nanotubes is defined as the quantity of SWCNTs relative to the metal catalysts and other carbon-like materials present (amorphous, graphitic, and C_{60} carbons). The strengths, limitations, and research needs for most of the commonly used techniques, e.g., TGA, TEM, SEM, Raman, and NIR were discussed. While reference materials will ultimately be useful, it was noted that the nanotube community must first agree on measurement and characterization methods. There was consensus that protocols for measurement techniques would be valuable even if they are incomplete. Details of some of the suggested protocols and their modifications are listed in the attached “Notes”.

Dispersion is defined as the distribution of nanotube bundles, the splitting of the bundles into individual tubes, and the agglomeration of SWCNTs in solvents or polymers. In macrodispersion, the focus is on dispersability (degree and ease of placing the nanotubes in suspension) and eliminating agglomerates. In nanodispersion, the focus is on debundling the SWCNT ropes. There was a suggestion that a standard dispersed liquid and a standard dispersed

solid be distributed to researchers willing to perform characterization so that methods can be compared. Variables could include composition, particle size, and dispersability. An agreement on a solvent for dispersion is also needed. Optical microscopy was conceded to be the primary technique for determining dispersion, but other techniques of value include SEM, UV/VIS Spectroscopy, and AFM. Details of some of the suggested protocols and their modifications are listed in the attached "Notes". Workshop participants unanimously agreed there was an urgent need for sonication methods research to determine the effects of time, frequency, and power of the ultra-sonicator on the SWCNT materials.

Participants agreed to support a special issue of the Journal of Nanoscience and Nanotechnology. The issue will feature a workshop summary, multi-authored review papers on specific techniques, and articles on research findings presented during the workshop. Possible topics are TGA, TEM, SEM, EDS, optical microscopy, UV/IR, light scattering, SANS, surface energy/area/tension, surfactants/dispersants, sonication, and magnetic techniques. In some instances NIST Recommended Practice Guides will also be written in order to provide more technical detail.

Potential future workshop topics were discussed. These include: 1) diameter, chirality, length, and types of nanotubes; 2) defects 3) surface chemistry and functionalization; 4) functional, e.g. electronic properties; 5) applications and performance measures; and 6) health and safety issues. **There was consensus that identification of measurement procedures relative to nanotube size and chirality would be particularly welcomed. NIST and NASA/JSC agreed to plan for such a workshop.**